

BEST AVAILABLE COPY

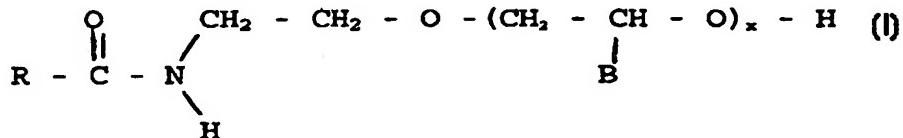
PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D 1/66	A1	(11) International Publication Number: WO 99/46356
		(43) International Publication Date: 16 September 1999 (16.09.99)
(21) International Application Number: PCT/US99/05177		(81) Designated States: AU, CA, CN, FI, JP, KR, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).
(22) International Filing Date: 10 March 1999 (10.03.99)		
(30) Priority Data: 09/038,736 11 March 1998 (11.03.98) US		Published <i>With international search report.</i>
(71) Applicant: MONA INDUSTRIES, INC. [US/US]; 76 East 24th Street, Paterson, NJ 07544 (US).		
(72) Inventors: PERELLA, James, E.; 49 Brookwood Drive, Mahwah, NJ 07430 (US). KOMOR, Joseph, A.; 15 Colonial Heights Drive, Ramsey, NJ 07446 (US). FOST, Dennis, L.; 208 N. Van Dien Avenue, Ridgewood, NJ 07450 (US). KATSTRA, Richard, D.; 151 Mt. Peter Road, Warwick, NY 10990 (US).		
(74) Agent: SCHOENBERG, Franklyn; Norman E. Lehrer, P.C., 1205 N. Kings Highway, Cherry Hill, NJ 08034 (US).		

(54) Title: IMPROVED ALKANOLAMIDES



(57) Abstract

Improved modified monoethanolamide compositions are provided which may be represented by formula (I), wherein: R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms; B is CH₃ or -CH₂ - CH₃; and x is from 1 to 6; wherein the modified monoethanolamide compositions, which are liquid at ambient temperatures or lower, exhibit surfactant properties substantially the same as those exhibited by diethanolamides such as foam stabilization and viscosity building as well as other desirable characteristics.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

IMPROVED ALKANOLAMIDES

5

Field of the Invention

The present invention relates to improved alkanolamide surfactants and, more particularly, to modified monoalkanolamides which are liquid at ambient temperatures 10 and to the method of making the same.

Background of the Invention

Nonionic surfactants are well known and have achieved fast growing commercial importance. They encompass 15 a broad range of compounds having a diverse range of structures and applications. One type of nonionic surfactants are alkanolamides that are condensates, for example, of fatty acids with alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA) and 20 monoisopropanolamine (MIPA), have been used in a variety of cosmetic, personal care, household and industrial formulations.

Alkanolamides are widely used in generally liquid systems such as liquid detergents and personal care products 25 as foam stabilizers, viscosity builders, solubilizers and the like, in metal working formulations as lubricants, viscosity control agents, corrosion inhibitors and in a variety of other applications. Alkanolamides utilized as components in such systems are ethanolamides and/or isopropanolamides such as monoethanolamides, diethanolamides and isopropanolamides in which the fatty acid acyl radical typically contains from 8 to 18 carbon atoms. Such dialkanolamides are typically liquid, while monoalkanolamides are solids having melting points of 40°C to 30 about 90°C. Heretofore, especially satisfactory alkanolamides have been diethanolamides such as those derived from coconut 35 oil mixed fatty acids or special fractions containing, for

instance, predominately C₁₂ to C₁₄ fatty acids. These alkanolamides are generally liquid in form which greatly simplifies their use.

Up to now, monoalkanolamides have not been available in liquid form which has limited their use in many applications. In recent years, because of regulatory concerns and restrictions, formulation trends have been shifting toward greater usage of monoalkanolamides; such products being solids, are more difficult to handle and are inconvenient to use in large scale production processes. Accordingly, it would be highly advantageous to be able to combine the performance characteristics of monoethanolamides with products having liquid physical characteristics at ambient conditions or lower.

Another well known basic group of nonionic surfactants are the polyoxylated derivatives, primarily represented by polyethoxylated and polypropoxylated compounds which are widely used as emulsifiers and detergents but do not provide the advantages of alkanolamides in connection with, for example, foam stabilization and viscosity building. Attempts have been made in the past to use ethylene oxide as an adduction agent for alkanolamides to modify the properties of the alkanolamides in a favorable manner including possibly reducing the congealing temperature of the monoethanolamides. While ethylene oxide adducts of alkanolamides were found to effect compositions with some modified properties, the amount of ethylene oxide needed to achieve physical property changes in the alkanolamides resulted in products which more closely resembled those exhibited by polyoxyethylene compounds, and the derivatives did not retain many of the characteristics of alkanolamides which were most desirable.

35

Summary of the Invention

It is accordingly an object of the present

invention to provide a process for preparing modified monoethanolamide surfactant compositions having a congealing 5 temperature less than about 25°C, that preferably are liquids at ambient temperature or lower (e.g. 25°C or lower), and that exhibit the foam stabilizing, viscosity building and other desirable surfactant characteristics of unmodified dialkanolamide and monoalkanolamide surfactants.

It is another object of the present invention to 10 provide a novel process for preparing an improved monoethanolamide surfactant composition by reacting a monoethanolamide surfactant composition having a congealing temperature of about 40°C or higher with an amount of 15 propylene or butylene oxide sufficient to prepare a modified monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower that substantially exhibits the surfactant characteristics of dialkanolamide surfactant compositions and the monoethanolamide reactant.

It is a further object of the present invention to 20 provide an improved modified monoethanolamide surfactant composition having a congealing temperature lower than about 20°C, that preferably is liquid at ambient temperature (about 25°C) or lower and that exhibits foam stabilizing, viscosity 25 building and the like characteristics of dialkanolamide and monoalkanolamide surfactants.

It is still a further object to provide cosmetic and other personal care preparations containing modified monoethanolamide surfactants having congealing temperatures 30 of about 20°C or lower.

It is yet another object of the present invention to provide metal working and other compositions for a variety of household and industrial applications containing modified monoethanolamide surfactants having congealing 35 temperatures of about 20°C or lower.

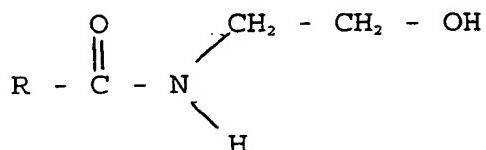
These and other objects will become apparent from the description to follow.

In accordance with the present invention there has now been discovered a novel process for preparing a

5

monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower which comprises:

a) providing a monoethanolamide composition
10 represented by the formula

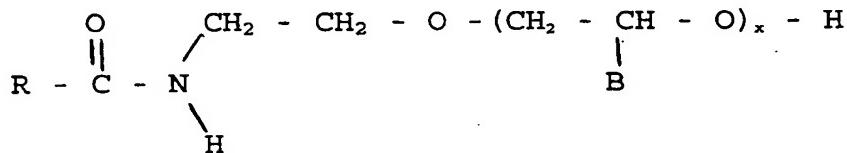


15 wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21 carbon atoms; and

b) reacting said monoethanolamide composition in
20 the presence of a suitable catalyst with an amount of
propylene oxide, butylene oxide or mixtures of the same
sufficient to form a modified monoethanolamide surfactant
composition that is substantially liquid at ambient
temperature, preferably having a congealing temperature of
25 about 20°C or lower, for a time sufficient for substantially
all of the alkylene oxide to react.

In another aspect of the present invention there are provided modified monoethanolamide surfactant compositions having a congealing temperature of about 20°C or lower represented by the formula



35

Wherein:

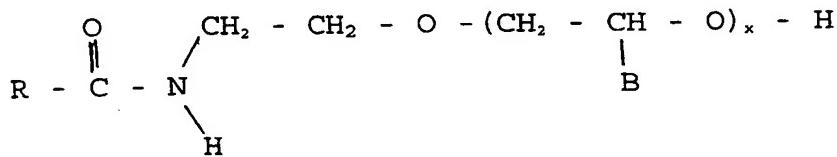
R is optionally substituted or unsubstituted,
branched or straight chain, saturated or unsaturated
5 hydrocarbon radical of 3-21 carbon atoms;

B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

10 The modified monoethanolamide compositions of the present invention which are substantially liquids at ambient temperature or lower, preferably, have a congealing temperature of about 20°C or, most preferably lower, surprisingly and unexpectedly generally exhibit most, if not 15 all, of the surfactant characteristics of dialkanolamide surfactant compositions such as the foam stabilization and viscosity building properties, as well as retaining substantially all the favorable properties of the monoethanolamides from which the compositions are prepared.

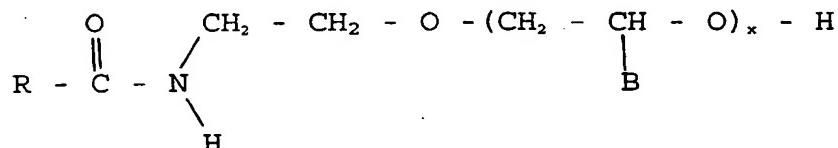
20 In yet another aspect of the present invention, there are provided cosmetic and personal care compositions which comprise at least 0.1% by weight of a modified monoethanolamide surfactant composition which is substantially a liquid at ambient temperature or lower that 25 may be represented by the formula



30 Wherein:
R is optionally substituted or unsubstituted,
branched or straight chain, saturated or
unsaturated hydrocarbon radical of 3-21 carbon
atoms;

B is CH₃ or -CH₂ - CH₃; and
x is from 1 to 6.

In a still further aspect of the present invention
5 there are provided metal working compositions and household
cleaning products which are preferably in liquid form
comprising at least 0.1% by weight of a modified
10 monoethanolamide surfactant composition which is
substantially a liquid at ambient temperature or lower that
may be represented by the formula



Wherein:

R is optionally substituted or unsubstituted,
branched or straight chain, saturated or
unsaturated hydrocarbon radical of 3-21 carbon
20 atoms;

B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

25

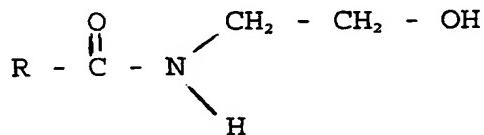
Description of the Preferred Embodiments

In accordance with the present invention,
materials and methods are provided which enhance the
properties of a well known and widely used class of nonionic
surfactant compositions making an easily handleable group
30 thereof readily and economically available for use in a
variety of cosmetic, personal care, household and industrial
applications.

The method of this invention applies to the
treatment of a monoethanolamide surfactant composition

generally having a congealing temperature of about 40°C or higher by reacting the normally solid monoethanolamide composition in the presence of a suitable catalyst, preferably potassium hydroxide or sodium alcoholate, with an amount of butylene oxide or preferably propylene oxide only sufficient to prepare a monoethanolamide derivative composition which is substantially liquid at ambient temperatures (about 25°C) and preferably has a congealing temperature of about 20°C or lower. The monoethanolamide derivative compositions which are prepared in accordance with the practice of the present invention exhibit surfactant properties such as foam stabilization and viscosity building as well as other desirable characteristics which are similar to those exhibited by dialkanolamides and are substantially the same as those provided by the unmodified monoethanolamide compositions. Moreover, the monoethanolamide derivative compositions of the present invention which are substantially liquid at ambient temperatures or lower, are not known to have any undesirable toxicological or environmental concerns.

In general, the method of the present invention can be accomplished by treating a monoethanolamide composition which has the formula



wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21, preferably 8- 18 carbon atoms;

with an amount of propylene oxide, butylene oxide or mixtures of the same in the presence of a suitable catalyst, such as potassium hydroxide, sodium alcoholate and

the like, that is only sufficient to form a monoethanolamide derivative composition which is liquid at ambient temperatures or lower and, preferably, substantially retains 5 the surfactant characteristics of the unmodified monoethanolamide composition.

The reaction of the monoethanolamide composition with propylene oxide, butylene oxide or mixtures of the same

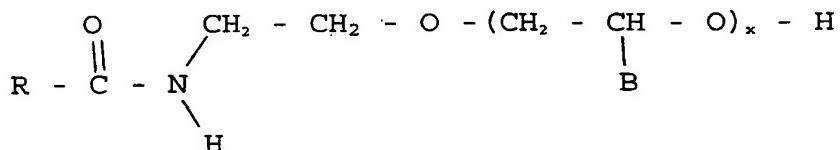
10 for adding one or more moles of alkylene oxide onto the alkanolamide in accordance with the invention can be carried out using any well known method. The degree of alkoxylation of the monoethanolamide composition being treated is important but may be varied depending upon the molecular 15 weight of the monoethanolamide composition and the degree of unsaturation in the fatty alkyl amide moiety as expressed by the iodine value thereof, generally by adding from about 1 to 6 moles, preferably from 1 to 4 moles, of propylene oxide, butylene oxide or mixtures thereof, per mole of 20 monoethanolamide. However, the number of moles of alkylene oxide used, as indicated, should be only the amount sufficient to produce a monoethanolamide derivative composition which is substantially liquid at ambient temperatures, and, preferably, has a congealing temperature 25 of about 20°C or, more preferably, lower.

The typically solid monoethanolamide compositions suitable for use in the preparation of modified monoethanolamide compositions of the present invention having a congealing temperature of about 20°C or preferably 30 lower in accordance with the practice of the present invention are well known and include those derived from substituted or unsubstituted, branched or straight chain, saturated or unsaturated fatty acids, esters or triglycerides with fatty alkyl amide moieties having from 3 to 21 carbon atoms, preferably having from 8 to 18 carbon 35 atoms. Examples of suitable fatty acids, esters or triglycerides from which the monoethanolamide compositions may be prepared include octanoic acid, decanoic acid, lauric

acid, myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, linolenic acid, methyl esters or glyceride esters of such acids or mixtures thereof as are found in coconut oil, palm oil, sunflower oil, soybean oil, rapeseed oil, castor oil, fish oil, tallow fat, milk fat, lard and other natural sources or may be of synthetic origin.

10

The modified monoethanolamide surfactant compositions of the present invention which are prepared in accordance with the practice of the present invention may be represented by the formula



Wherein:

20 R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21, preferably, 8-18 carbon atoms;

25 B is -CH₃ or -CH₂ - CH₃; and

x is from 1 to 6, preferably from 1 to 4.

As indicated, the monoethanolamide compositions which are typically high temperature melting waxy solids that are converted into the monoethanolamide derivatives of the present invention may be prepared in any suitable manner and numerous processes for their productions are well known. The modified monoethanolamide surfactant compositions of the present invention, which are liquids at ambient temperatures and, preferably, have a congealing temperature of about 20°C or lower, retain the generally useful and desirable surfactant and other properties of the monoethanolamide

compositions from which they are prepared as well as those exhibited by diethanolamide compositions that are well known and widely used.

5 The degree of alkoxylation, that is, the number of groupings "x" of the above formula, may be varied but only within narrow limits. Monoethanolamide derivative compositions of the invention which are adducts of only a sufficient amount of butylene oxide or, preferably,

10 propylene oxide per mole of monoethanolamide, generally from 1-6 moles, preferably from 1-4 moles, of alkylene oxide, surprisingly and unexpectedly are liquids at ambient temperatures and preferably have a congealing temperature of
15 about 20°C or lower while advantageously retaining the desirable surfactant characteristics of the unmodified monoethanolamide compositions as well as displaying the surfactant properties of diethanolamides. Depending on the molecular weight and degree of unsaturation of the fatty
20 alkyl amide moiety of the monoethanolamide composition as expressed by the iodine value (I.V.) thereof, the monoethanolamide derivative compositions of the present invention will contain an amount of propylene oxide to monoethanolamide derivative composition in the range from at
25 least 22% by weight to about 57% by weight, or an amount of butylene oxide to monoethanolamide derivative composition in the range from about 40% by weight to 60% by weight, although in the case of monoethanolamide compositions containing saturated high molecular weight amide moieties
30 (iodine values of about 0), the amount of propylene oxide and/or butylene oxide to monoethanolamide derivative of about 60% by weight or higher may prove to be desirable.

 The novel modified monoethanolamide compositions of the invention display many of the well known properties of diethanolamides such as foam stabilization and viscosity building as well as other desirable properties such as emulsification of oil based materials, solubilization of fragrances and hair colorants and dyes, wetting of natural

and synthetic fibers, compatibility with anionic surfactants and detergents and the like while exhibiting congealing temperatures which are substantially lower than those of the
5 unmodified monoethanolamide compositions, thus making them easy to formulate into a wide range of cosmetic, personal care, household and industrial systems. Surprisingly and unexpectedly it has been found that while alkoxylation of monoethanolamides with controlled, small amounts of
10 propylene oxide or butylene oxide in accordance with the practice of the invention provides monoethanolamide derivatives which are liquid at ambient temperatures or lower and retain all or most of the desirable surfactant characteristics of the unmodified monoethanolamides and of
15 diethanolamides, reactions which involve treating typically solid monoethanolamide compositions with 5 or more moles of ethylene oxide in the presence of a suitable catalyst are useful in forming derivatives thereof which are flowable solids or gels at ambient temperatures (20-25°C) but the
20 monoethanolamide derivatives thus formed, significantly lack many of the desirable alkanolamide surfactant properties such as foam stabilization and viscosity building.

It is therefore a further aspect of the invention to use the novel compositions of the invention, for example,
25 in cosmetic preparations and the like, especially in preparations for hair care and skin treatment. In this connection, personal care formulations can be shampoos, hair colorants, hair conditioners, bath products and skin treatment cremes and lotions. Other formulations where the
30 properties of the novel compositions of the invention can be utilized include make-up cremes, sunscreens, lipstick, pressed powders, skin-toners, antiperspirants and the like. Shampoos to which the novel modified monoethanolamide compositions of the invention have been added in amounts of
35 from 0.1 to 10 percent by weight or greater and which contain up to about 30 weight percent of substances with detergent activity, in addition to water and possibly other ingredients, result in compositions having desired foam

stability and viscosity building as well as many other desirable characteristics.

Conventional additives such as perfumes, preservatives, complexing agents, opacifiers, luster developing agents and the like may be added to any of the above mentioned personal care products.

The novel modified monoethanolamide compositions may also be added to a wide variety of home care and industrial formulations wherein their usefulness as detergents, metal working and lubricating agents, emulsifiers, anticorrosion agents for metal products and various other liquid and/or water based personal care, home care and industrial compositions wherein diethanolamide and unmodified monoethanolamide compositions have been found useful. The preparation of specific compositions of the invention is illustrated by the following examples which are provided herein for purposes of illustration only and are not intended to limit the scope thereof.

20

Example 1

A three-necked flask fitted with an agitator, thermometer, dry-ice condenser, heating mantle and addition funnel is charged with 125 grams of a solid commercially available coconut monoethanolamide having the tradename MONAMID CMA, which is prepared from coconut oil and monoethanolamine. The solid monoethanolamide (congealing temperature 63°C; Iodine Value (I.V.)- 9) is melted at 70 - 80°C, 0.9 grams of 85% potassium hydroxide catalyst are added and the mixture is stirred until homogeneous. The temperature of the reaction mixture is raised to 140-150°C and 58 grams of propylene oxide is added dropwise over a period of 1.5 hours. The reaction mixture is stirred at 140-150°C for an additional hour after which it is cooled to 25°C. The propoxylated monoethanolamide product with a propylene oxide content of 31.5% by weight is a clear amber liquid having a congealing temperature of -1°C.

Example 2

Using the reaction vessel of example 1, 125 grams
5 of a solid commercially available coconut monoethanolamide
having the tradename MONAMID CMA-A, which is prepared from
coconut fatty acid and monoethanolamine, is reacted with 58
grams of propylene oxide using the procedure of example 1. A
clear amber liquid product is prepared having a congealing
10 temperature of 0°C.

Example 3

A) Using the reaction vessel and procedure of
example 1, a propoxylated monoethanolamide is prepared from
15 125 grams of the coconut monoethanolamide material of
example 1 except that only 29 grams of propylene oxide is
added to the heated monoethanolamide reactant. After
completion of the reaction, the product with a propylene
oxide content of 18.7% by weight is cooled to 25°C and a
20 nonclear pasty liquid results.

B) Using the reaction vessel and procedure of
example 1, a propoxylated monoethanolamide is prepared from
125 grams of the coconut monoethanolamide material of
example 1 except that 174.3 grams of propylene oxide is
25 added to the heated monoethanolamide reaction mixture. After
completion of the reaction and cooling, a reaction product
with a propylene oxide content of 58.2% by weight which is a
clear amber liquid is obtained.

C) Using the reaction vessel and procedure of
30 example 1, a propoxylated monoethanolamide is prepared from
125 grams of the coconut monoethanolamide material of
example 1 except that 232.4 grams of propylene oxide is
added to the heated monoethanolamide reaction mixture. After
35 completion of the reaction and cooling, a clear dark amber
liquid reaction product is obtained having a propylene oxide
content of 65.0% by weight.

Example 4

A) Using the reaction vessel of example 1, 192.6
5 grams of a soya monoethanolamide (tan solid - congealing temperature 45°C; Iodine Value - 130) prepared from soybean oil and monoethanolamine is charged into the reactor and heated at 70-80°C together with 1.5 grams of flake 85% potassium hydroxide. The mixture is agitated until
10 homogeneous and the temperature is then raised to 140-150°C. While maintaining the temperature with agitation, 104.6 grams of propylene oxide is added dropwise over a period of 2.5 hours. The reaction mixture is stirred for an additional hour at 140-150°C and then cooled to 25°C. After cooling, the
15 reaction product with a propylene oxide content of 35.0% is a clear amber liquid having a congealing temperature of -1°C.

B) The reaction of A) above is run except that only 69.76 grams of propylene oxide is added dropwise to the molten soya monoethanolamide reactant. The resultant product
20 having a propylene oxide content of 26.4% is a paste at 25°C.

Example 5

Using the reaction vessel of example 1, 230.1 grams of a caprylic/capric monoethanolamide (I.V. - 0)
25 prepared from a C₈/C₁₀ triglyceride and monoethanolamine is charged into the reactor with 1.5 grams of 85% flake potassium hydroxide and heated at 70-80°C with agitation until a homogeneous mixture is formed. The temperature of the reaction mixture is then raised to 140-150°C and 68.4
30 grams of propylene oxide is added dropwise over a period of 1.5 hours. The reaction mixture is stirred for another hour at a temperature of 140-150°C. The reaction product with a propylene oxide of 22.8% by weight is a clear amber liquid at 25°C.

Example 6

A) Using the reaction vessel of example 1, 125
5 grams of the solid coco monoethanolamide of example 1 is charged into the reaction vessel and melted at 70-80°C at which time 0.9 grams of 85% potassium hydroxide is added and the mixture is stirred until homogeneous. The temperature of the reaction mixture is raised to 140-150°C and 108 grams of
10 butylene oxide are added dropwise to the reaction mixture with stirring over a period of 1.5 hours. The reaction mixture is maintained at 140-150°C with stirring for an additional hour, after which it is cooled to 25°C. The reaction product with a butylene oxide content of 46.2% is a
15 clear amber liquid.

B) The reaction of A) above is run except that only 72 grams of butylene oxide are added dropwise to the coco alkanolamide reaction mixture over a period of 2.5 hours. After cooling, the reaction product having a butylene
20 content of 36.4% is a solid.

C) The reaction of A) above is run except that 180 grams of butylene oxide are added dropwise to the coco alkanolamide reaction mixture over a period 2.5 hours. After cooling, the reaction product with a butylene oxide content
25 of 59% is a clear amber liquid.

Example 7

The propoxylated monoethanolamide derivative compositions of examples 1, 4A and 5 are used in this
30 example. Foam stabilization properties of various modified monoethanolamide compositions are evaluated and the properties effected by the monoethanolamide derivative compositions (all having congealing temperatures less than 0°C) of the present invention are compared with those
35 obtained with a commercial diethanolamide composition, an unmodified monoethanolamide (solid with a congealing

temperature of 63°C) and a commercial monoethanolamide reacted with 5 moles of ethylene oxide (congealing temperature 25°C). The results of the tests are reported below in Table 1.

Table 1Ross Miles Foam Test (mm of Foam)

	<u>Sample Tested</u>	<u>At 0 Minutes</u>	<u>After 1 Minute</u>	<u>After 5 Minutes</u>
10	Example 1 Comp.	230	201	197
15	Unmodified Coconut monoethanolamide MONAMID CMA	231	206	201
20	Example 4A Comp.	236	212	206
25	Example 5 Comp.	233	208	203
30	Coconut Diethanolamide	234	204	201
	Coconut Monoethanolamide + 5 moles of Ethylene Oxide	135	119	15

The test is run according to ASTM Method D1173-53 with 19% of Sodium Lauryl Sulfate + 1% test amide. 0.1% total active in 0 ppm water hardness temperature = 25°C.

As can be seen, the monoethanolamide derivative compositions with low levels of propoxylation, which are liquids, exhibit excellent foam stabilization properties when blended with an anionic surfactant, such as sodium lauryl sulfate, substantially the same as the foam stabilization characteristics of coconut diethanolamide and unmodified coconut monoethanolamide. In contrast thereto, an ethylene oxide adduct of the coconut monoethanolamide is not liquid and does not stabilize foam.

Example 8

The propoxylated monoethanolamide derivative compositions of examples 1, 4A and 5 are used in this example. Viscosity building properties of various monoethanolamide derivative compositions of the invention are evaluated and compared with the properties effected by a commercial diethanolamide composition (congealing temperature -4°C), an unmodified monoethanolamide (solid-congealing temperature 63°C) and a commercial monoethanolamide adducted with 5 moles of ethylene oxide (congealing temperature 25°C). The results are reported below in Table 2.

15

Table 2Viscosity Comparisons of Sodium Lauryl Sulfate Blends

	<u>Sample Tested</u>	<u>Viscosity (cPs) 1% Added NaCl</u>
20	Example 1 Comp.	283
	Unmodified Coconut monoethanolamide MONAMID CMA	625
25	Example 4A Comp.	120
	Example 5 Comp.	130
	Coconut Diethanolamide	173
30	Coconut Monoethanolamide + 5 moles of Ethylene Oxide	45
35	Test Formulation: 19% of Sodium Lauryl Sulfate active + 1% test sample.	

While the propoxylated monoethanolamide compositions (Examples 1, 4A and 5 Compositions) exhibit somewhat reduced viscosity building characteristics compared

to an unmodified coconut monoethanolamide composition, they are approximately equivalent in viscosity building to a coconut diethanolamide composition and are clearly superior
5 to the ethylene oxide adduct of a coconut monoethanolamide.

Example 9

The alkoxylated monoethanolamide derivative compositions of examples 1, 2, 3B) and 3C) are used in this
10 example. Prototype hair shampoo formulations are prepared using 30% active sodium lauryl sulfate and 3% active monoethanolamide derivative compositions. A shampoo sample is prepared for comparison purposes from a solid commercially available fatty acid derived monoethanolamide
15 having the Trade Name MONAMID CMA-A. The various shampoo samples are evaluated for Ross-Miles Foam and Viscosity and the results are reported in Table 3, below.

Table 3

		<u>Sample Tested</u>				
		CMA-A	Comp 1	Comp 2	Comp 3B	Comp 3C
	Ross-Miles Foam (mm) initial	185	205	208	180	177
25	1 min.	170	179	180	155	152
	5 min.	165	172	171	147	142
	Viscosity (cP)					
	No Salt	25	16	16	4	6
30	1% NaCl	6,200	440	2,375	7.5	5

As can be seen, the monoethanolamide derivative compositions with low levels of propoxylation (example 1 and 2 compositions), which are liquids, exhibit excellent foam
35 stabilization properties when blended with an anionic surfactant, such as sodium lauryl sulfate, substantially the same as the foam stabilization characteristics of unmodified coconut monoethanolamide which is not a liquid. In contrast thereto, the coconut monoethanolamide derivative compositions with high levels of propylene oxide, example
40

3B) and 3C) compositions, do not stabilize foam as effectively. Moreover, the propoxylated monoethanolamide compositions with low levels of propylene oxide (Example 1 5 and 2 Compositions) which are liquids, exhibit significant viscosity building characteristics, whereas the propoxylated monoethanolamide compositions with high level of propylene oxide, example 3B) and 3C) compositions, exhibit poor viscosity building characteristics.

10

Example 10

The alkoxyolated monoethanolamide derivative compositions of examples 1 and 2 are used in this example. Cotton wetting by the monoethanolamide derivative 15 compositions of the invention are evaluated and compared to the wetting characteristic of the unmodified monoethanolamide composition of example 1. The results are reported in Table 4, below.

As can be seen, the wetting characteristics of the 20 liquid modified monoethanolamide compositions of the invention, which are readily soluble and/or dispersible in water, are clearly superior to the wetting characteristics of a solid unmodified monoethanolamide composition.

Table 4Draves Cotton Skein Wetting Test

5 Concentration: 0.1% active in deionized water*

Temperature: 25°C

Method: AATCC 17-1994

Cotton Skein Wetting Time

	Unmodified Coconut Monoethanolamide <u>Triglyceride-Derived</u>	Composition of <u>Example 1</u>	Composition of <u>Example 2</u>
10	Test terminated with no wetting after 35 minutes	Wets at 39.0 seconds	Wets at 39.5 seconds
15			

*Note: The propoxylated alkanolamide compositions of examples 1 and 2 are noted to be more easily dispersible and soluble than the unmodified alkanolamide composition.

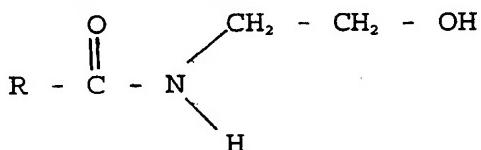
20 it will be evident from the above that there are other embodiments and methods, which while not expressly described above, are clearly within the scope and spirit of the invention. The description above is therefore intended
25 to be exemplary only and the scope of this invention is to be limited solely by the appended claims.

21

WHAT IS CLAIMED IS:

1. A method for preparing a modified monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower which comprises:

- 5 a) providing a monoethanolamide composition represented by the formula



10 wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3 - 21 carbon atoms; and

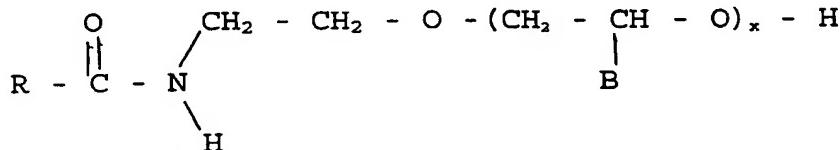
- b) reacting said monoethanolamide composition in
15 the presence of a suitable catalyst with an amount of
propylene oxide, butylene oxide or mixtures of the same,
only sufficient to form a modified monoethanolamide
surfactant composition that is substantially liquid at
ambient temperature or lower.

20 2. The method as claimed in claim 1, wherein said monoethanolamide composition is reacted with from 1 to 6 moles of propylene oxide, butylene oxide or mixtures of the same.

3. The method as claimed in claim 1, wherein said
25 monoethanolamide composition is reacted with from 1 to about
4 moles of propylene oxide.

4. The method as claimed in claim 1, wherein said monoethanolamide composition to be reacted is a solid having a congealing temperature of at least about 40°C.

30 5. A modified monoethanolamide surfactant composition having a congealing temperature of about 20°C or lower of the formula



35

Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

5 B is CH₃ or -CH₂ - CH₃; and

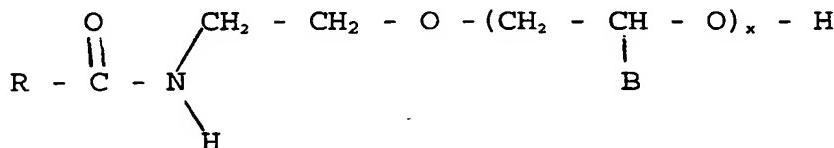
x is from 1 to 6;

said modified monoethanolamide composition being substantially liquid at ambient temperature or lower, and
10 being suitable to exhibit foam stabilization and viscosity building properties.

6. The modified monoethanolamide surfactant composition as claimed in claim 5, wherein x is from 1 to 4.

7. The modified monoethanolamide surfactant
15 composition as claimed in claim 5, wherein B is a CH₃ group.

8. Cosmetic, personal care and household use compositions which comprise at least 0.1% by weight of a modified monoethanolamide surfactant composition which is substantially a liquid at ambient temperature or lower of
20 the formula



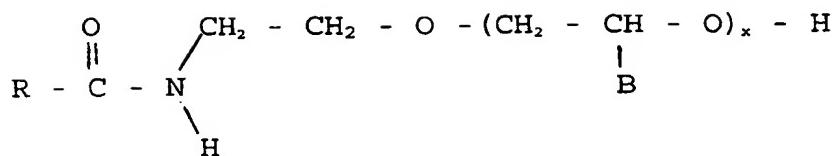
25 Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

30 B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

9. Metal working and industrial use compositions which are in liquid form comprising at least 0.1% by weight of a modified monoethanolamide surfactant composition which
35 is substantially a liquid at ambient temperature or lower of the formula



5

Wherein:

R is optionally substituted or unsubstituted, branched or straight chain, saturated or unsaturated hydrocarbon radical of 3-21 carbon atoms;

10 B is CH₃ or -CH₂ - CH₃; and

x is from 1 to 6.

INTERNATIONAL SEARCH REPORT

International application No. PCT/US99/05177

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C11D 1/66
US CL :510/421, 433, 499, 505

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 510/421, 433, 499, 505

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

STN STRUCTURE SEARCH

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 8-337,560 A (KAWAKEN FINE CHEMICAL CO., LTD) 24 December 1996. Schemes on pages 9 and 12, column 3-column 5, table on page 15.	1-9
A	JP 9-67,325 A (KAWAKEN FINE CHEMICAL CO, INC.) 11 March 1997, entire document.	
A	JP 4-136,289 A (DAIICHI KOGYOSEIYAKU CO. LTD.) 11 May 1992, entire document.	
A	JP 61-64,322 A (NIPPON OILS AND FATS CO. LTD.) 04 February 1986, entire document.	
A	DE 2,643,804 A1 (BASF AG) 06 April 1978, entire document.	

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means		
P document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

27 APRIL 1999

Date of mailing of the international search report

19 MAY 1999

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

JOHN R. HARDEE

BRIAN A. HARDEN
PARALEGAL SPECIALIST
GROUP 100

Telephone No. (703) 308-0661

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.